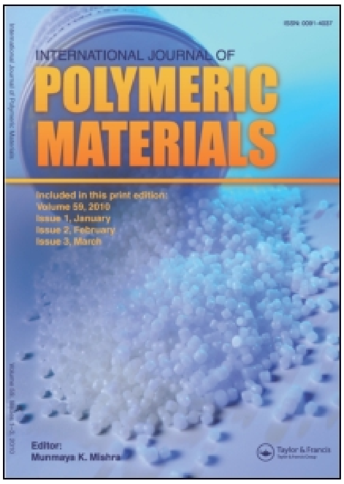

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Fatigue Behavior of Epoxy and Polyester Resins

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The behavior of an epoxy (Epon 828) and polyester resin (Paraplex P43) have been studied when subjected to cyclic fatigue stresses. A rotating beam fatigue machine was used to apply the stresses which varied from compression to tension (mean stress = 0). The surface temperature of the specimens was recorded by using an infrared temperature detector and the temperature rise was studied as a function of cyclic frequency and stress level. Fatigue life of the materials was studied as a function of cyclic frequency and the influence of moisture on fatigue life was also studied. It has been determined that the temperature rise during fatigue is critical in determining the life of the material. Fractography studies are also discussed and characteristic fracture surface features will be indicated.

1 INTRODUCTION

The increasing engineering applications of thermosetting materials as matrices for composite materials have created a need for better understanding of the fatigue fracture behavior of these materials. In recent years, a great amount of research has been done on the fatigue properties of fiber reinforced plastics but comparatively little is known about the fatigue of polymeric materials used for composite matrices. Broutman and Sahu[1] have shown that the fatigue breakdown in fibrous composites is quite dependent upon the matrix material; thus, the matrix can determine the fatigue life of the composite. The fatigue mechanisms for the polymeric materials are complex in nature because of their molecular structure, temperature and environmental sensitivity, hysteresis and viscoelastic response. It has been shown that thermoplastic polymeric materials can fail under cyclic load[2,3] and an appreciable specimen

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temperature rise can take place during fatigue tests.[4,5] It has been reported that the fatigue mechanism of polymers is controlled by the internal damping of the materials and the failure occurs with a sharp temperature increase. Recently, it has been shown that cyclic fatigue failure can also occur at very low cyclic frequency and at low amplitudes of stress in which case the temperature increase of the specimen[6] during the fatigue tests is very small.

In the present study an attempt has been made to determine the role of the specimen temperature increase during the fatigue tests. Flexural fatigue of an epoxy resin and a polyester resin was studied using a rotating beam fatigue machine. The effect of providing water and air cooling to the surface of the specimen during the test has been studied with respect to fatigue life. The effect of environment on fatigue life of these materials has also been studied. Experiments were carried out to determine if the fatigue of these materials is cycle dependent or time dependent. Finally, the sensitivity of these materials to cyclic load frequency is demonstrated by carrying out the tests at various frequencies.

2 EXPERIMENTAL PROCEDURE

The two materials selected for this study were an epoxy (Epon 828)† and a polyester (Paraplex P43)‡. The procedure suggested by the material manufacturer was followed in the preparation of the casting mixtures. For the epoxy resin, metaphenylene diamine (MPDA) curing agent was used (14.6 per cent by weight). The resin was mixed under vacuum to minimize the entrapment of air bubbles. It was then spun in a centrifuge at about 1500 rpm to expel minute air bubbles which might have been entrapped during the mixing operation. The mixture was then poured slowly into cylindrical teflon molds which were preheated to 200°F.

For polyester resin specimens, benzoyl peroxide (1 per cent by weight) was mixed in styrene monomer (7 per cent by weight) and this was then mixed with the polyester resin. Mixing and casting procedures similar to that for epoxy resin specimens were followed.

The cast bars which were $\frac{11}{16}''$ diameter and $5\frac{1}{2}''$ long were then machined to the final shape of the specimens. Figure 1 shows the design of the fatigue specimen. For flexural strength tests (3 point bending), the specimens were prepared by casting the resin in the form of a flat plate.

An Automation Industries Model RBF 25 fatigue machine (25 in. lb capacity) was used foı the fatigue tests. The surface temperature of the specimen was recorded by using an infrared temperature detector (Ircon model CH 34L).

†Tradename, Shell Chemical Co.
‡Tradename, Rohm and Haas Co.

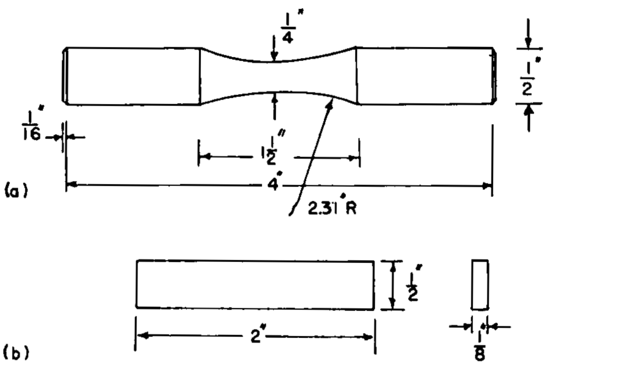

FIGURE 1 Test specimens

3 RESULTS AND DISCUSSION

1 Fatigue Lives

The actual fatigue results obtained are presented in Figures 2 and 3. Nearly 100 specimens have been tested to establish these curves. For the epoxy resin, only a small stress range could be evaluated because of the low modulus of the material and resulting large deflection of the specimen when loaded. Thus an upper limit is placed on the stress which can be applied to the specimen.

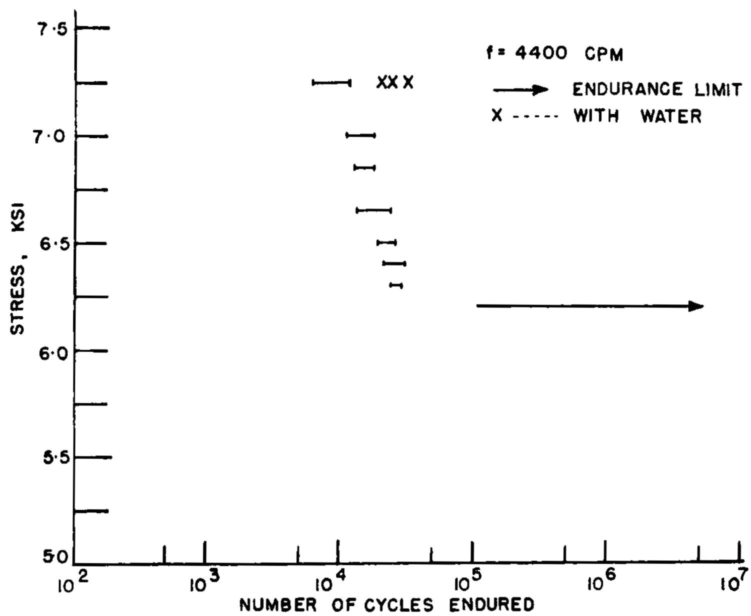

FIGURE 2 S–N curve for epoxy resin

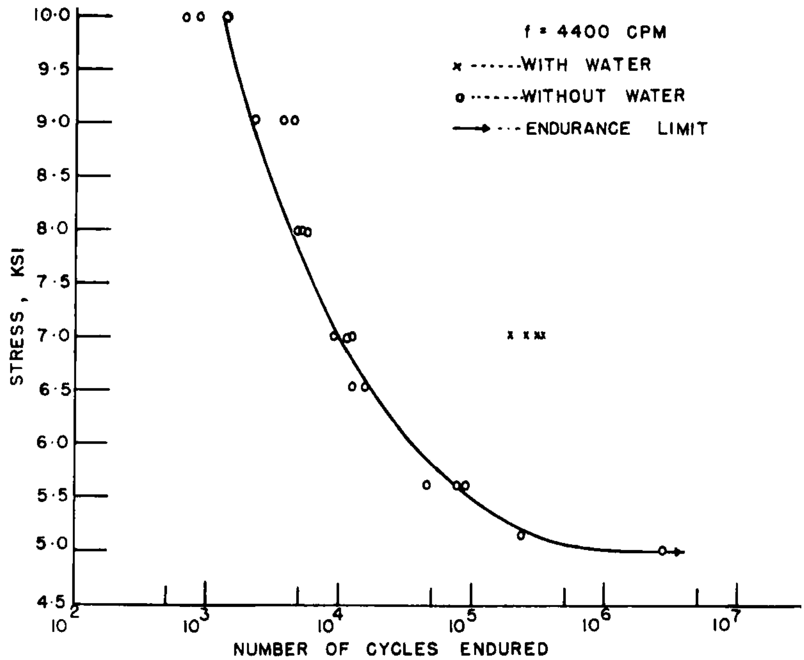

FIGURE 3 S–N curve for polyester resin

Due to this limitation, only scatter bands have been shown in the fatigue curve for the epoxy resin. The fatigue curves for these two materials resemble fatigue curves for metals and it appears that endurance limits exist for these materials. For each material 5 specimens were tested at a stress level below the endurance stress limit and none of them failed after 10^6 cycles were applied. These curves have been established only for one cyclic frequency and the fatigue life data are quite sensitive to the cyclic frequency as will be discussed later.

2 Temperature Rise During Fatigue

The temperature rise of a specimen (as a result of hysteresis loss) has been studied as a function of cyclic frequency and stress level. Previously, Dally and Broutman[7] studied the temperatuie rise in fibrous composites as a function of cyclic frequency and stress level. The surface temperature versus number of fatigue cycles for the epoxy resin at two different levels are shown in Figure 4. Surface temperatures as high as 190°F were measured at the time of specimen fracture.

The temperature rise curves for the polyester resin at various levels are shown in Figure 5. These curves are somewhat different from those observed

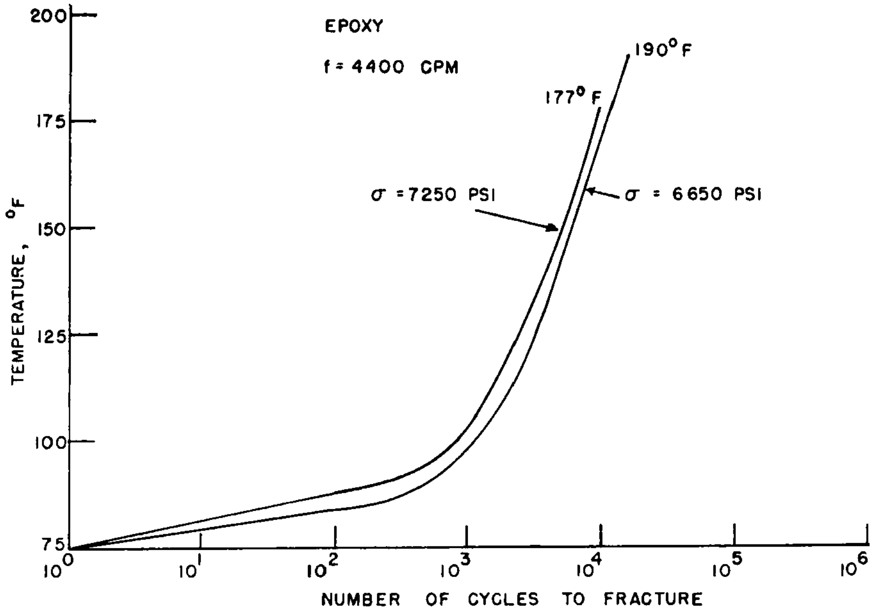

FIGURE 4 Temperature rise as a function of fatigue cycles

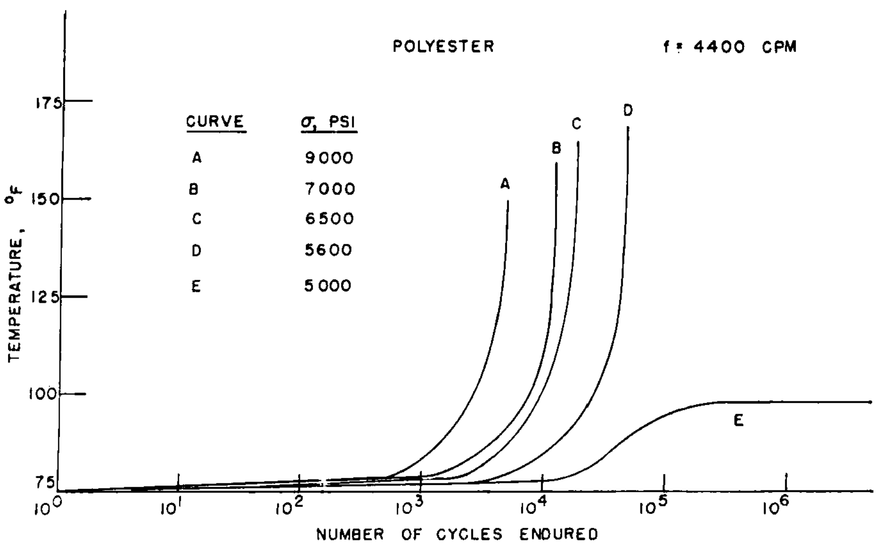

FIGURE 5 Temperature rise as a function of fatigue cycles

for epoxy resin. For the polyester resin, a very rapid increase in surface temperature is observed in the last few cycles of its life.

The increase in surface temperature of specimens during fatigue testing is attributed to the hysteresis loss in the material. To understand the relatively sharp temperature rise of a polyester fatigue specimen during the last few cycles of its life, specific hysteresis loss data were determined from cyclic flexural tests on an Instron at various temperatures. These data for the epoxy and polyester resins are presented in Table Ia and Table Ib respectively. It is observed from these results that the specific hysteresis loss increases with increasing temperature for the polyester resin. Hence it can be inferred that as the temperature of the specimen increases during the fatigue test, an increasing amount of hysteresis heating takes place during the last few cycles. For epoxy resin, the specific hysteresis loss is not sensitive to temperature, and this results in more uniform hysteresis heating during each cycle.

It is observed from these curves that the surface temperature at fracture increases with decreasing stress. This is expected because the specimen endures more cycles to failure at lower stresses and this increase in number of cycles results in more hysteresis heating of the specimen.

TABLE I

Specific hysteresis loss for epoxy resin and polyester resin

a. Epoxy resin

Stress applied psi	$\frac{\Delta W}{W} \times 10$			
	73°F	115°F	162°F	207°F
7000	1.85	1.56	1.49	—
10,500	1.65	1.089	1.082	1.032
14,000	1.43	1.31	1.13	—
17,500	1.49	1.52	—	—

b. Polyester resin

Stress applied psi	$\frac{\Delta W}{W} \times 10$		
	73°F	115°F	162°F
3500	2.34	2.80	4.17
7000	1.80	2.36	4.06
10,500	1.60	1.82	—

ΔW – Hysteresis loss.
W – Total work done (area under stress strain curve).

The surface temperature at the endurance limit approaches a steady state value at the respective test frequency. The steady state temperatures for the

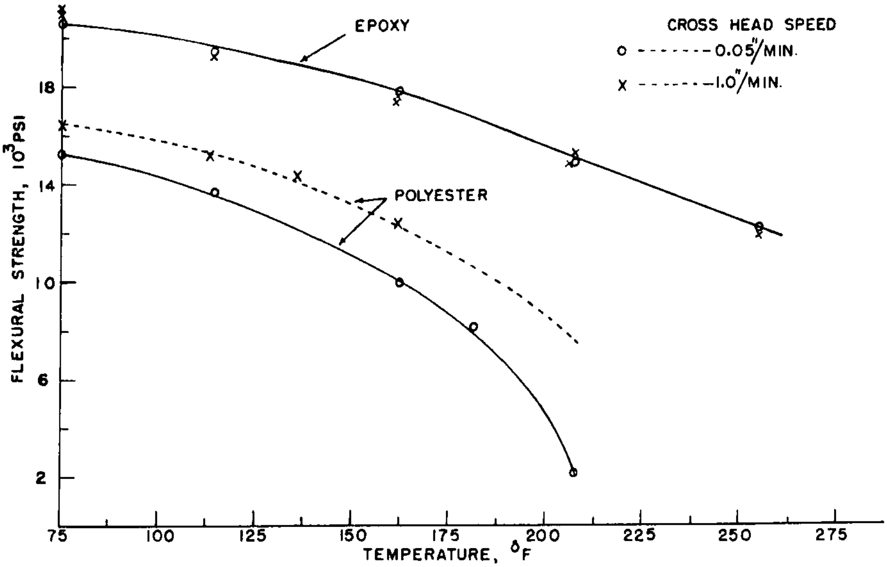

FIGURE 6 Variation of flexural strength with temperature

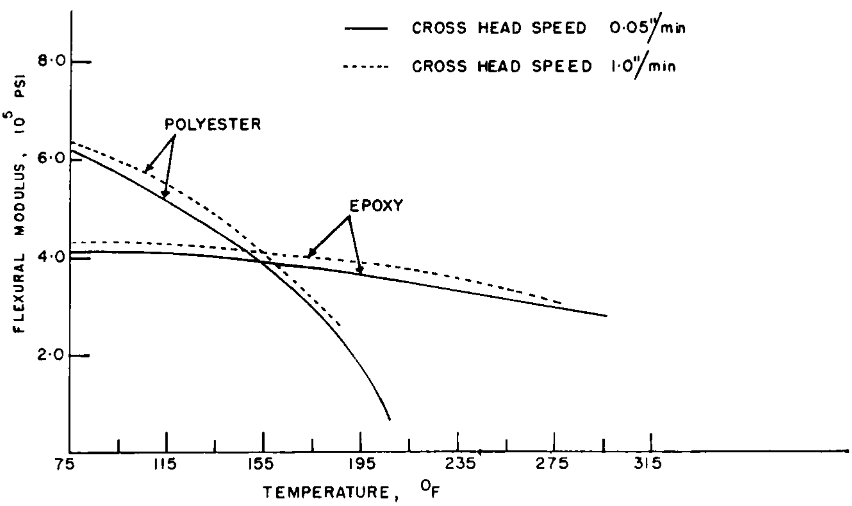

FIGURE 7 Variation of flexural modulus with temperature

epoxy resin and polyester resin are found to be 142°F and 95°F, respectively (at 4400 cpm). The temperature rise of a specimen influences the fatigue life by lowering the ultimate strength and stiffness of the material. These properties are dependent upon temperature and Figure 6 and Figure 7 show the flexural strength and flexural modulus at various temperatures. It is observed from these curves that the flexural strength and modulus of the polyester resin are greatly reduced as the temperature is increased. For the epoxy resin, these properties are less altered by the same change in temperature. Thus, if the temperature of the specimen rises during fatigue tests, the flexural properties are changed significantly which in turn should influence the fatigue life.

To further determine the effect of temperature rise on fatigue life of these materials, tests were carried out with air cooling provided to the surface of the specimen. The results for epoxy and polyester resin are presented in Table II. For epoxy resin, the surface temperature is maintained at 140°F and a significant increase in fatigue life is noted. For the polyester resin, the surface temperature of the specimen is maintained at 85°F and the fatigue life is increased by a factor of nearly 5 over the fatigue life established without cooling.

The increase in fatigue life of these materials in the presence of a coolant is further demonstrated by tests performed with the specimens immersed in water. A chamber is placed around the specimen in the fatigue machine and water is circulated through the chamber. The composite effect of water is not only to reduce the temperature of the specimen but also to provide a different environment for the specimen. The results for epoxy and polyester resin are indicated in Figures 2 and 3, respectively.

TABLE II

Effect of air cooling on fatigue life

	No cooling		Air cooling	
Material	Number of cycles endured	Surface temperature at fracture	Number of cycles endured	Surface temperature at fracture
Epoxy				
$\sigma = 7250$ psi	10,000	177°F	13,500	140°F
$f = 4400$ cpm	10,000	179°F	16,000	140°F
	9600	180°F		
Polyester				
$\sigma = 7000$ psi	13,000	158°F	44,200	85°F
$f = 4400$ cpm	10,300	140°F	57,000	85°F
	6600	110°F		

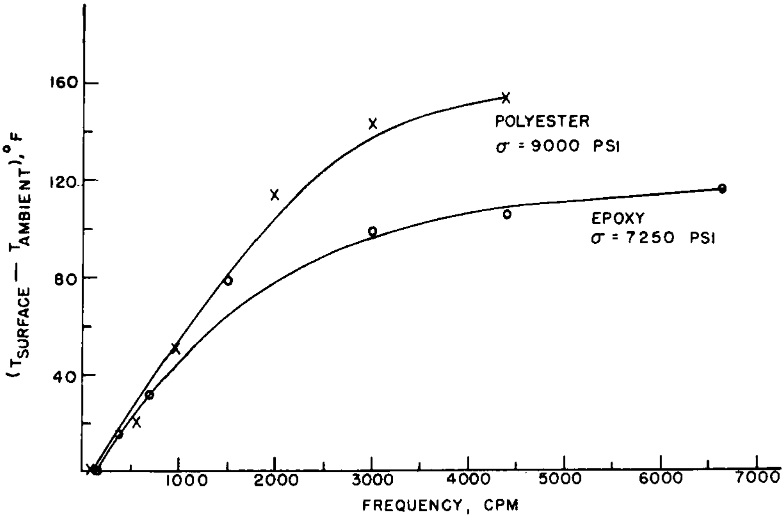

FIGURE 8 Temperature increase versus frequency

3 Effect of Cyclic Frequency

As mentioned earlier, the maximum surface temperature of the resins is influenced by cyclic test frequency. The frequency versus maximum surface temperature curves for the epoxy and polyester resin are shown in Figure 8. The curve for the epoxy resin indicates that at a stress level of 7250 psi, the surface temperature will not increase above ambient unless the cyclic frequency is greater than 150 cycles per minute. For the polyester resin, there is no increase in the surface temperature of the fatigue specimen at frequencies below 100 cycles per minute for a stress level of 9000 psi. These results would vary with the shape and size of the fatigue specimens.

Table III shows the number of fatigue cycles endured by polyester specimens at different cyclic frequencies for a stress level of 9000 psi. Table IV presents similar results for the epoxy resin at a stress level of 7250 psi. These results confirm the fact that fatigue life increases with decreasing cyclic frequency. Note that polyester specimens fail, at a cyclic frequency of 100 cpm, although there is no increase in surface temperature of the specimen. This demonstrates that fatigue fracture in this material can occur even without a temperature increase and the corresponding reduction of strength which accompanies the temperature increase.

4 Effect of Environment on Fatigue Life

The effect of atmospheric environment on cyclic fatigue of these materials has been studied. The method employed to isolate the specimens from water

TABLE III

Fatigue data for polyester resin at various frequencies
$\sigma = 9000$ psi

Frequency in cycles/min	Maximum number of cycles endured
6600	—
4400	3600
3000	4600
2000	5200
1000	7600
600	14,400
100	40,400

TABLE IV

Fatigue data for epoxy resin at various frequencies
$\sigma = 7250$ psi

Frequency in cycles/min	Maximum number of cycles endured
6600	9300
4400	10,000
3000	10,700
1500	80,000
700	No failure (1×10^6)
320	No failure (5×10^5)
150	No failure (3×10^5)

vapour in the atmosphere was to coat the specimens before they were fatigued. The epoxy and the polyester specimens were held at 100°C and 50°C respectively for 2 hours and then coated with a graphite coating. The coating was applied in the oven so that the specimens could not absorb any water from the atmosphere during the coating operation. Finally, a silicone wax was applied to the surface over the graphite coating. These specimens were then cycled at one stress level and frequency and the number of cycles to failure are compared with those of uncoated specimens subjected to the same stress level and frequency. The probability of failure is plotted versus the number of cycles to failure for the epoxy and polyester resins in Figures 9 and 10, respectively. The life of epoxy resin is increased by the coating. For epoxy resin, the loss in weight after heating was measured to be 0.103% of the original weight. It is thought that the water absorbed in the epoxy resin acts as a plasticizer and thus reduces the flexural strength. Thus, the increase in fatigue life of coated specimens can be attributed to the expected increased flexural strength due to removal of water. For polyester resin, there is no increase in

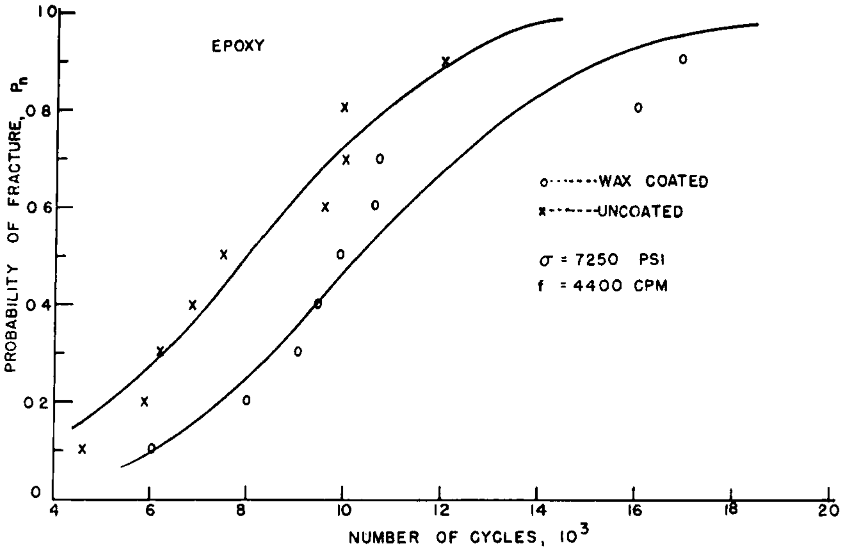

FIGURE 9 Effect of wax coating on probability of fatigue fracture

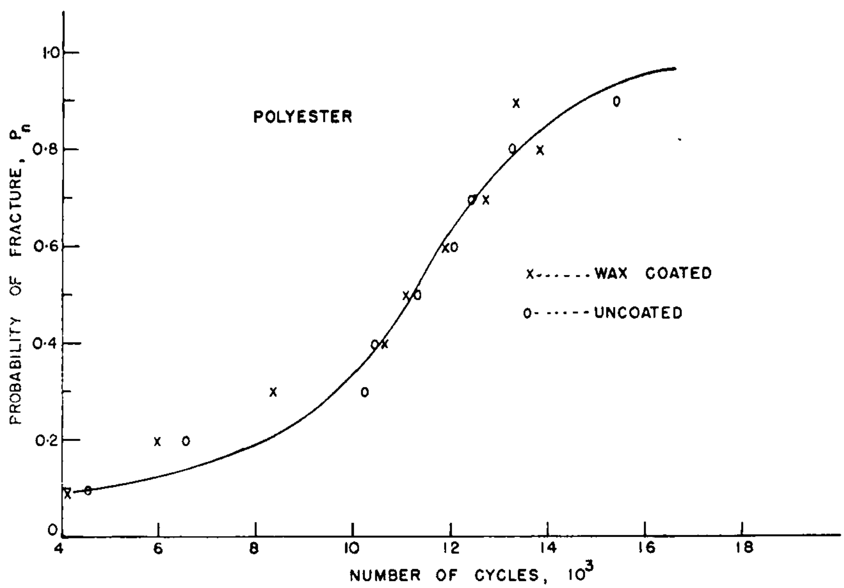

FIGURE 10 Effect of wax coating on probability of fatigue fracture

fatigue life with the protective coating. However, as already stated, these specimens could only be heated to 50°C as they distorted too much above this temperature. This temperature is not sufficient to drive off water absorbed in the specimen or absorbed on the surface. The loss in weight after heating was found to be only 0.016% of the original weight of the specimen.

A water environment surrounding the specimen increases the fatigue life of these materials as discussed earlier. Although diffusion of water into the specimen takes place along the surface, the cooling effect provided by the water is much more pronounced and primarily responsible for the increase in fatigue life which was observed and discussed earlier.

5 Cycle Dependent Fatigue Fracture

From the above discussions it is clear that the epoxy and polyester resins fail under cyclic stress but this does not necessarily prove that the fatigue failure is cycle dependent. It could very well be time dependent. To clarify this point, specimens were subjected to static bending stresses for a time which far exceeded the time to failure under the cyclic stress. The results are listed in Table V. These results show that specimens subjected to static bending stresses do not break in this time period. As there was no temperature rise of the specimen during these tests, it was thought that the failure could be time dependent at the higher temperatures which occur during the cyclic fatigue tests. To show that this is not the case, the specimens were heated during a static fatigue test to the temperatures which actually occurred in the cyclic fatigue test at that particular stress level. The results shown in Table 5, indicate that no failure occurs even after periods of time equivalent to 80 times the time periods in the cyclic fatigue test. Thus it can be said that the fatigue of the epoxy and polyester resins is truly cycle dependent and not time dependent. In other words the cyclic stress produces a cyclic crack growth which leads to fracture. An additional reason for the difference in the time to failure between rotating bending tests and static tests is that the entire specimen volume is subjected to the maximum tensile stress in the case of the rotating beam test. Thus, the probability of a flaw being exposed to the stress for the rotating beam is greater than for the static beam.

6 Intermittent Fatigue

Intermittent fatigue tests were performed for these materials and the results are shown in Table VI. For the polyester resin, the applied stress of 9000 psi was removed as soon as the temperature of the surface reached 95°F (which is the steady state temperature of the material as defined earlier). The machine was stopped and the stress was reapplied when the specimen cooled to room temperature. This procedure was repeated till failure. It is observed that the

TABLE V

Static fatigue data

Material	Time to failure in cyclic fatigue test $f = 4400$ cpm	Static fatigue at room temperature	Static fatigue at higher temperature	
		Time under static bending stress	Surface temperature	Time under static bending stress
Epoxy				
$\sigma = 7250$ psi	2 min 15 sec	160 hrs (No failure)	175°F	180 min (No failure)
$\sigma = 7000$ psi	2 min 30 sec	—	175°F	180 min (No failure)
Polyester				
$\sigma = 9000$ psi	43 sec	160 hrs (No failure)	155°F	180 min (No failure)
$\sigma = 7000$ psi	2 min 50 sec	—	155°F	180 min (No failure)

specimen fails but the fatigue life is increased considerably as compared to the fatigue life for continuous loading. Similar results are obtained by removing the stress when the surface temperature reaches 80°F.

By repeating this procedure at an applied stress of 7000 psi (removing the stress when temperature of the specimen reached 80°F) the specimen did not fail at 1×10^5 cycles. Similar results are obtained when the applied stress is removed at 90°F.

For the epoxy resin, with a similar test and a cut-off temperature of 140°F, the specimen does not fail up to 1×10^5 cycles at a stress level of 7250 psi. For continuous application of cycles the specimen fails at 10,000 cycles.

These results show that cumulative damage laws will not be valid for these materials when temperature rises occur. These results also show that no permanent damage in the material occurs under cyclic stress for the first few

TABLE VI

Intermittent fatigue results

Material	Continuous fatigue life at $f = 4400$ cpm	Cut-off temperature†	No. of cycles at cut-off temperature	Total number of cycles endured
Polyester				
$\sigma = 9000$ psi	3600	95°F	2000	16,000
		90°F	1500	12,000
		80°F	1000	33,200
$\sigma = 7000$ psi	13,000	80°F	3000	1×10^5 (No failure)
		90°F	6000	1×10^5 (No failure)
Epoxy				
$\sigma = 7250$ psi	10,000	140°F	2400	100,000 (No failure)

†Denotes the temperature at which applied stress is removed during the tests.

cycles and the fatigue cracks do not propagate during this period. The fatigue cracks will start propagating only at a particular temperature of the specimen which will depend on the applied stress and the stress concentration of a pre-existing flow.

It is also inferred from the above tests that fatigue life is increased by performing the intermittent fatigue tests particularly when the specimen temperature is maintained below the steady state temperature of the material.

7 Fatigue Fracture Surfaces

A fracture surface of polyester fatigue specimen is shown in Figure 11. The appearance of the fracture surface indicates a brittle type of fracture and the fracture initiates at a flaw near the surface of the specimen. The relatively smooth surface area (mirror area) around the crack origin is typical of many polymers and indicates a region of slow crack growth.[8–11] Fatigue striations are not observed at this stress level (10,000 psi).

Figure 12 shows the fracture surface of a specimen cycled at a stress level of 6500 psi. This fracture surface is different from the surface shown in Figure 11. The crack origin is located at the surface and brittle fatigue striations near the crack origin are distinctly visible. Figure 13 shows the enlarged view of the striation zone. Each striation is caused by a single load cycle and represents the successive position of the crack front after each load cycle. These striations are similar to those observed on fracture surfaces of some metals.[12–14] Another feature in common with the fracture surfaces of metals is the tongue shaped development of the crack front as shown in Figure 12.

Figure 14 shows the various parabolic markings seen on the fracture surface near the edge. These markings are caused by the initiation of secondary fractures in advance of the crack front due to the high stresses extending ahead of the oncoming main crack. The secondary fracture origins may be inclusions, voids or inhomogeneities in the material.

Figure 15 shows the striations formed near the edge of the specimen. These striations look different from the striations formed near the crack origin. The striations in Figure 15 are almost evenly spaced and are bowed in the direction of crack propagation. A possible explanation available in the literature for this bowing phenomenon is the existence of a plane stress condition near the surface of the specimen and a plane strain condition inside the specimen. The bowing of these striations at tear lines is also a common feature and is clearly shown in Figure 15. These striations have been observed in metals and certain polymers and are known as ductile striations. The formation of ductile striations in a brittle polyester can be attributed to the effect of the temperature rise during cyclic fatigue tests resulting in increased ductility of the material.

FIGURE 11 Fracture surface showing fracture origin well inside the surface ($\times 19$)
$\sigma = 10{,}000$ psi, $f = 4400$ cpm

Figure 16 shows a fracture surface for a specimen cycled at $\alpha = 6000$ psi. A well defined tongue shaped crack front is clearly seen here. Figure 17 shows the crack front at higher magnification. The brittle striations near the crack origin are very closely spaced but the spacing increases with each load cycle. It is observed that brittle striations are continuous at tear lines whereas ductile striations bow at tear lines as explained earlier.

In Figure 18 the fracture initiates from a flaw well inside the specimen away from the boundaries where the applied bending stress is lower than at the boundary. The stress concentration at this flaw site causes the fracture to initiate from that point and there are no brittle striations present. It has been observed that brittle striations either do not appear or are less pronounced when the fracture originates at an inclusion inside the material. This may be because very high stresses due to a stress concentration can occur at inclusions even if the applied stress is low. This results in very few or no striations as the brittle striations are favored at low stresses, as the stable crack can grow and reach a greater length before unstable crack propagation occurs. For example,

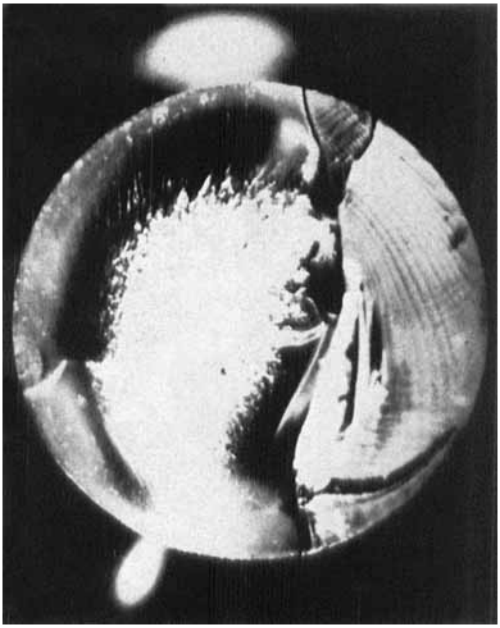

FIGURE 12 Fracture surface showing striations and tongue shaped crack front (×19) $\sigma = 6500$ psi, $f = 4400$ cpm

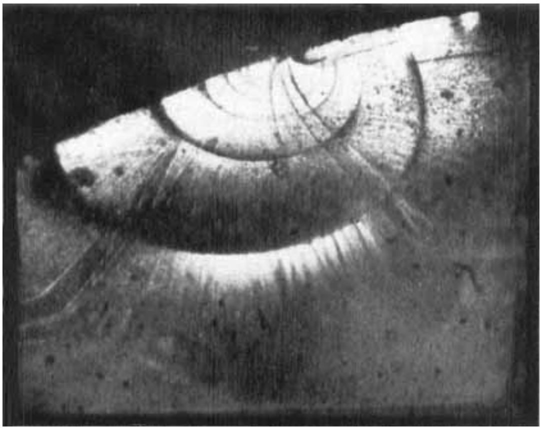

FIGURE 13 Enlarged view of striation zone (×50)

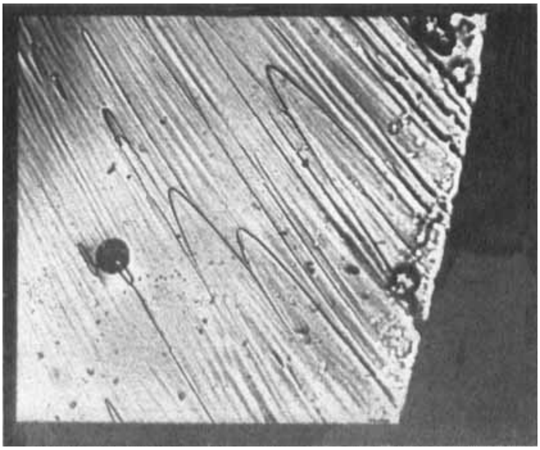

FIGURE 14 Parabolic markings near the edge ($\times 200$)

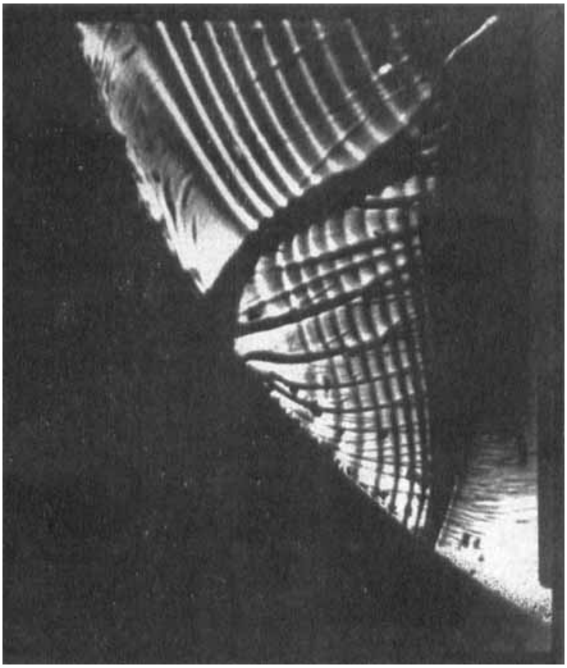

FIGURE 15 Ductile striations near the edge ($\times 50$)

Figure 19 shows a specimen cycled at approximately the same stress level as the specimen of Figure 18, but note the brittle striations in Figure 19.

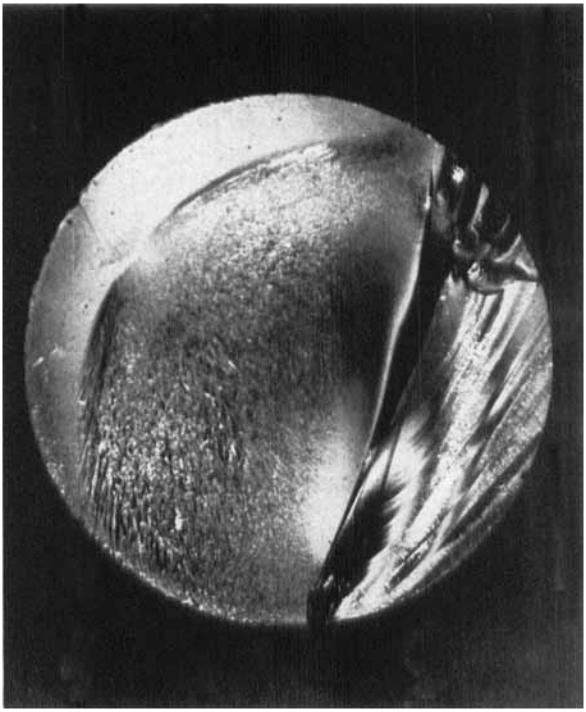

FIGURE 16 Fracture origin at the surface and tongue shaped crack front ($\times 19$)
$\sigma = 6000$ psi, $f = 4400$ cpm

The spacing of the brittle striations is not constant. An increase in crack length is associated with increased spacing. The tendency for striations to become more widely spaced as the crack grows is due to an increase in applied stress caused by progressively reducing the area of the uncracked material.

Figure 20 shows a plot of striations spacing versus distance of striations from the crack origin for several specimens cycled at various stress levels. A linear relationship exists between the crack length and striation spacing. The results are based on the last five brittle striations in the mirror area. The linear relationship is expected because the crack velocity is progressively increasing as the uncracked area is decreasing. This also supports the view that mirror area is the area of stable crack growth. Striations are not observed on specimens cycled at stress levels greater than 8000 psi regardless of the cyclic frequency. Thus, the formation of striations is only dependent upon the stress level and occur at the lower applied stresses.

The fracture surfaces of epoxy resin specimens are similar to the polyesters. In Figure 21 are shown the brittle striations observed in the mirror region at a

FIGURE 17 Enlarged view of crack front and brittle striations in mirror region (×200)

FIGURE 18 Crack origin at an inclusion well inside the surface (×19)
$\sigma = 5600$ psi, $f = 4400$ cpm

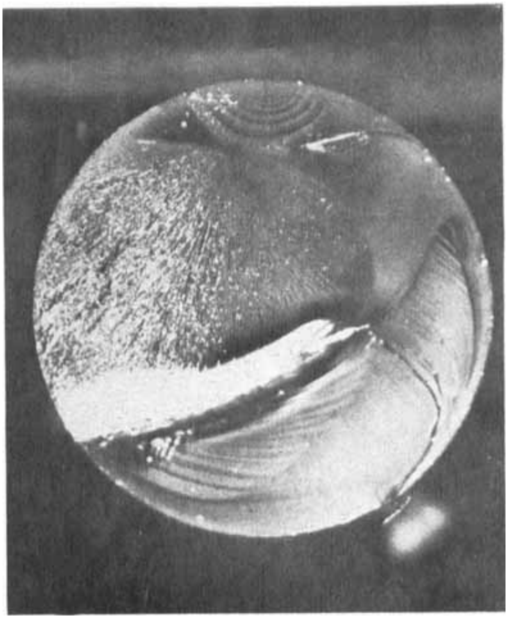

FIGURE 19 Crack origin at a surface flaw ($\times 19$)

stress level of 7250 psi. The fracture origins for the epoxy resins were primarily located at the surface and can be attributed to machining or polishing flaws. As in the case of polyester, the mirror region size increases with decreasing stress level.

4 CONCLUSIONS

The degradation in strength of epoxy and polyester resins occurs more rapidly under cyclic loading than under static loading, since a rotating beam fails in less time than a statically located beam. These materials exhibit fatigue behavior similar to metals in the sense that the number of fatigue cycles endured increases with decreasing applied stress. An endurance limit exists for the epoxy and the polyester resin. An appreciable amount of hysteresis heating takes place in these materials and the temperature of the specimen increases during the fatigue tests. The increase in temperature of a fatigue specimen is a function of specimen geometry, cyclic frequency and applied stress. The maximum surface temperature at fracture increases with increasing frequency. Below a given frequency there is no increase in surface temperature of a fatigue specimen. The cyclic frequencies at which surface temperature starts rising above ambient temperature are 100 cpm and 150 cpm for the polyester ($\sigma = 9000$ psi) and epoxy ($\sigma = 7250$ psi) resin specimens ($d = 0.25$ inches) respectively. At higher applied stresses, a specimen endures a

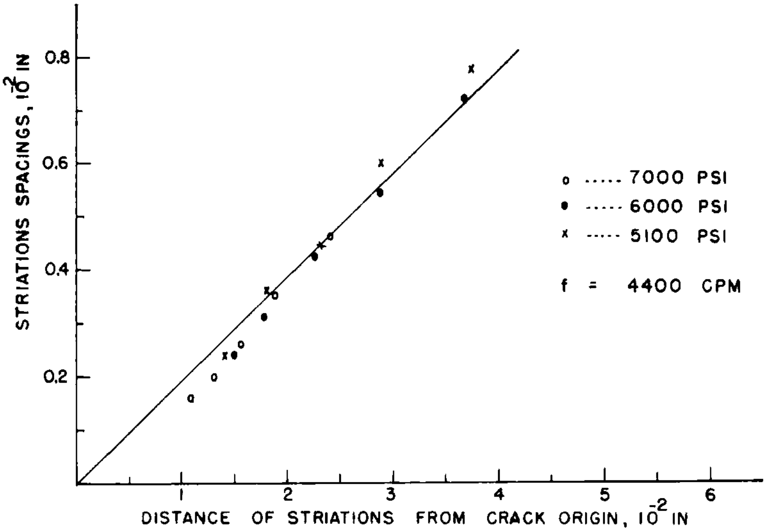

FIGURE 20 Striations spacing versus crack length

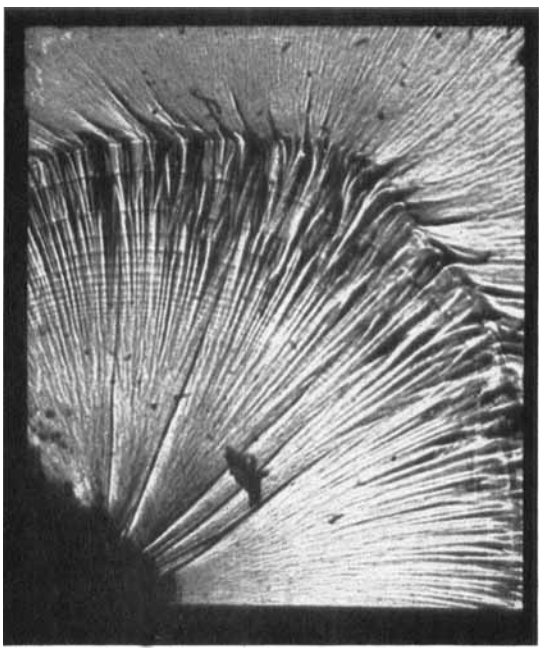

FIGURE 21 Brittle striations in mirror region of epoxy resin specimen (×200)

fewer number of fatigue cycles which results in lower surface temperature of fracture.

In the presence of a coolant like air or water, the fatigue life of the epoxy and polyester resin is increased. The fatigue life of these materials is sensitive to test environment. For the epoxy resin, the fatigue life in a laboratory atmosphere is less than that with a protective surface coating. The presence of water environment increases the fatigue life of the epoxy and polyester resin considerably.

The fatigue cracks in the epoxy and polyester resin originate at pre-existing flaws which include machining marks on the surface, air bubbles and inclusions introduced during the casting process. For the epoxy resin, the fatigue crack invariably starts at a surface flaw for the stress range considered in this study. For the polyester resin, fracture has been observed to initiate at surface flaws as well as at internal inclusions or air bubbles. At low stresses, fracture usually starts at a surface flaw.

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